Effect of Metal Ion-doping on Water Splitting Performance of Metal Oxide Electrocatalysts

Synopsis in Candidature for the Degree of

Doctor of Philosophy in Physics

Submitted by

Mr. Ahmed Hasan Hasan Al-naggar

Under the guidance of

Prof. Rajaram Sakharam Mane

School of Physical Science

Submitted to

Swami Ramanand Teerth Marathawada University, Nanded

2019-20

1. Introduction

Global environmental problems are crucial international issues and have been increasing with the growth of world economy. Due to the reality that fossil fuels are running out as well as the global warming, development of environmental-friendly and CO_2 emission-free fuel is an urgent issue [1,2]. Hydrogen/Oxygen is considered as an ideal fuel for the future. Hydrogen/Oxygen fuel can be produced from clean and renewable energy sources and, thus, its life cycle becomes clean and renewable energy. In recent years, the technology for the evolution of hydrogen from electrolysis using solar cells has been extensively studied, but this methodology meets its limitation when it comes to the capital cost of cells itself. On the other hand, the use of photocatalyst is considered to have the satisfactory potential to produce hydrogen at low cost and could become a viable substitute for fossil fuel energy [3-5]. A fundamental problem related with the use of solar and wind energies is their inability to operate independently from demand, since their unscheduled intermittent supply often mismatches the grid power demands. Thus, the development and application of systems for the efficient storage of excess electricity is entailed [6,7] Electrocatalytic technologies can play a crucial role for the indirect storage of surplus renewable energy via the conversion of electricity to chemical energy [8]. Among them, water electrolysis is a promising alternative compared to electricity storage using batteries [9]. Hydrogen production via electrolysis using renewable energy sources amounts to only 4% of today's hydrogen production, mainly due to economic factors (i.e. lack of widely available renewable energy systems of low cost, high capital cost, high energy input required) [10]. This picture is going to change in the near future and increase in the use of renewable energy sources is expected, since the European Energy Directive has set the target for covering 14% of the energy needs by renewable energy sources by 2020. Hydrogen production by water electrolysis using renewable energy sources is expected to play a key role in the scenario of a green energy economy [11]. Clear advantages of this method is the high efficiency and the high purity of the produced hydrogen, which is of great importance for its subsequent conversion to electricity using low temperature polymer electrolyte fuel cells [12]. Furthermore, high purity oxygen is a valuable by-product of water electrolysis. Its utilization both in medical care and in chemical industry (blast furnaces, electric furnaces and glass melting, gasification) could lead to substantial decrease in the nominal cost of water electrolysis [13]. Currently, due to the need of decreasing our usage of fossil fuels, a revitalized interest is seen where improving the

catalytic efficiency of water splitting reaction in a sustainable and cheap way is one of the major challenges.

1.1 Water Electrolysis

Eco-friendly and cost-effective way of producing energy for the urgent requirement, it needs to be improved to make it cheaper and eco-friendlier. Although there are several types of fuel cells, the hydrogen (H_2) and oxygen (O_2) fuel cell is the one with zero carbon emission and water as the only by-product. And also photo electrochemical water splitting is also a promising technology for solar hydrogen production to build a sustainable, renewable and clean energy economy [14,15]. So great attention has been focused on hydrogen as potential energy vector and on the use of water splitting technology as clean and renewable means to generate hydrogen using solar energy and electrochemical energy. Water electrolysis produce hydrogen and oxygen from water by the hydrogen evolution reaction (HER) and the oxygen evolution reaction (OER). At the anode, water is split into oxygen gas with accompanying protons and electrons [16]. At the cathode hydrogen gas evolve by recombination of the protons and electrons. By far, this energy conversion technology has attracted extensive attention due to its high energy conversion efficiency, negligible environment pollution and potentially wide range of application. Due to that various catalyst are developed or synthesized that works in electrochemical and photo-catalysts. And also, water splitting activity of synthesized metal oxide/chalcogenide carried out with oxygen evolution reaction (OER) and hydrogen evolution reaction (HER) [17]. Surprisingly excellent catalytic activity and remarkable long-term stability and durability toward the HER in both acidic and basic media was exhibits by highly dense self-supported NiS−Ni9S8−NiSe Nano rod arrays

Figure 1. shows a schematic illustration of a water electrolysis setup composed of an anode, a cathode, an external power supply, and a conductive electrolyte. Under the applied voltage, hydrogen and oxygen are produced on the cathode and anode, respectively. At room temperature and using neutral water, the electrolysis of water proceeds slowly because pure water does not have a good electric conductivity with only 10^{-7} M of ion concentration. Therefore, in general, acid, alkaline or salts are added to improve the conductivity of the electrolyte. In addition, different geometries and solutions are used to ensure that the redox reactions occur close to each other but still keep the recombination low. According to different types of electrolytes used and approaches to separate the reactions, water

electrolysis can be divided into three main classifications; alkaline water electrolysis, polymer electrolyte membrane (PEM) water electrolysis, and solid oxide electrolyte (SOE) water electrolysis.

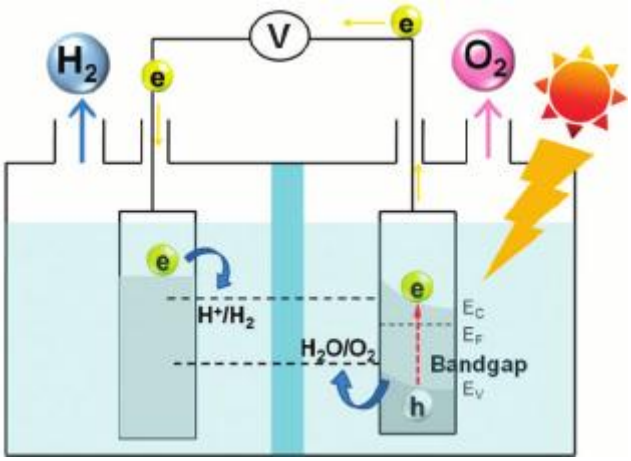

Fig.1: Schematic illustration of a water electrolysis cell where oxidation of oxygen in water occurs at the anode and reduction of hydrogen occurs at the cathode.

2. Literature review

(1) Zhou *et al*. the reported that Flower-like NiFe Oxide Nanosheets on Ni Foam as Efficient Bifunctional Electrocatalysts for the Overall Water Splitting [18].

Synthesized NiFe oxide exhibits excellent catalytic activity that surpasses the activity of the RuO_2 benchmark catalyst, when tested as an electrocatalyst for the overall water splitting reaction. More importantly, the optimized NiFe oxide electrode possesses excellent OER activity in 1 M KOH with small overpotentials of 255 and 280 mV at 10 and 50 mA cm^2, respectively. When employed as a stable bifunctional catalyst to split water, this electrode achieved a current density of 10 mA cm^{-2} at a cell voltage of 1.59 V. This work presents a highly promising candidate for use as an electrode material and represents promising progress in its practical utilization and comprehensive industrialization.

(2) Zhou *et al*. the studies of "Exceptional Performance of Hierarchical Ni–Fe (hydr) oxide@ NiCu Electrocatalysts for Water Splitting." Advanced Materials 31.8 (2019): 1806769. [19].

In this study earth-abundant transition metals (i.e., nickel, iron, and copper), 3D hierarchical nanoarchitectures, consisting of ultrathin Ni–Fe layered-double-hydroxide (Ni–Fe LDH) nanosheets or porous Ni–Fe oxides (NiFeOx) assembled to a metallic NiCu alloy, were delicately constructed. In alkaline solution, the as-prepared Ni–Fe LDH@NiCu possesses outstanding OER activity, achieving a current density of 10 mA cm^{-2}at an overpotential of 218 mV, which was smaller than that of RuO_2 catalyst (249 mV**).** The remarkable activity of the Ni–Fe LDH@NiCu and NiFeOx@NiCu was further demonstrated by a 1.5 V solar-panel-powered electrolyzer, resulting in current densities of 10 and 50 mA cm^{-2} at overpotentials of 293 and 506 mV, respectively. Such performance rendered the as-prepared materials as the best bifunctional electrocatalysts so far.

(3) Wan *et al*. The reported that "Hierarchical Porous Ni3S4 with Enriched High-Valence Ni Sites as a Robust Electrocatalyst for Efficient Oxygen Evolution Reaction." Advanced Functional Materials 29.18 (2019): 1900315. [20].

In this study, a highly active and stable, meso–macro hierarchical porous Ni_3S_4 architecture, enriched in Ni^{3+} was designed as an advanced electrocatalyst for OER. The obtained Ni_3S_4 architectures exhibited a relatively low overpotential of 257 mV at 10 mA cm^{-2} and 300 mV at 50 mA cm^{-2}. Additionally, this Ni_3S_4 catalyst showed excellent long-term stability (no degradation after 300 h at 50 mA cm^{-2}). The outstanding OER performance was due to the high concentration of Ni^{3+} and the meso–macro hierarchical porous structure. This work provided a new strategy for the rational design of highly active OER electrocatalysts with high valence Ni^{3+} and hierarchical porous architectures.

(4) **Shinde *et al*. The reported that "Electrocatalytic Water Splitting through the Ni_xS_y Self-Grown Superstructures Obtained via a Wet Chemical Sulfurization Process." ACS Omega 4.4 (2019): 6486-6491.**[21]

Water-splitting applications of chemically stable self-grown nickel sulfide (Ni_xS_y) electrocatalysts of different nanostructures including rods, flakes, buds, petals, etc., synthesized by a hydrothermal method on a three-dimensional Ni foam (NiF) in the presence of different sulfurion precursors, e.g., thioacetamide, sodium thiosulfate, thiourea, and sodium sulphide were reported the as-obtained Ni_xS_y electrocatalyst from sodium thiosulfate endowed overpotential of 200 mV. The oxygen evolution overpotential resulted of Ni_xS_y

electrocatalysts are comparable or superior to those reported previously for other self-grown Ni_xS_y superstructure morphologies.

(5) Qin *et al.* "A Tannic Acid–Derived N-, P-Codoped Carbon-Supported Iron-Based Nanocomposite as an Advanced Trifunctional Electrocatalyst for the Overall Water Splitting Cells and Zinc–Air Batteries." Advanced Energy Materials 9.5 (2019): 1803312. [22].

A trifunctional electrocatalyst composed of FePx nanoparticles and Fe–N–C moiety supported on the N-, P-codoped carbon (NPC) was masterly synthesized by facile one-pot pyrolysis of the mixture of tannic acid, ferrous chloride, and sodium hydrogen phosphate. This work showed offers a new strategy to design and synthesized highly effective multifunctional electrocatalysts using cheaper tannic acid derived carbon as supported applied in electrochemical energy devices.

(6) Nath *et al.* reported "MOF-templated cobalt nanoparticles embedded in nitrogen-doped porous carbon: a bifunctional electrocatalyst for overall water splitting." Nanoscale Advances (2019). [23].

The synthesis of nitrogen-enriched core–shell structured cobalt–carbon nanoparticles dispersed on graphene sheets were used for HER performances in both acidic and basic media. they have achieve These catalysts exhibited excellent durability and HER activities with onset overpotentials as low as ∼70 mV in both acidic (0.5 M H2SO4) and alkaline (0.1 M NaOH) electrolytes, and the overpotentials needed to deliver 10 mA cm^{-2} were determined to be 265 mV in acid and 337 mV in base, further demonstrating their potential to replace Pt-based catalysts.

(7) Lv *et al.* reported "Homogeneously Distributed NiFe Alloy Nanoparticles on 3D Carbon Fiber Network as a Bifunctional Electrocatalyst for Overall Water Splitting." ChemElectroChem 6.9 (2019): 2497-2502. [24].

An effective and facile strategy to prepare non-noble nickel-iron (NiFe) alloy nanoparticles (NPs) decorated on oxidized carbon cloth (OCC) as a bifunctional electrocatalyst for overall water splitting was high lighted.

(8) Lin *et al.* reported "Defect-Rich Heterogeneous MoS2/NiS2 Nanosheets Electrocatalysts for Efficient Overall Water Splitting." Advanced Science (2019): 1900246. [25].

In this reported free-standing and defect-rich heterogeneous MoS_2/NiS_2 nanosheets for overall water splitting were designed that showed the enhanced electrocatalytic performance as bifunctional electrocatalysts for overall water splitting application.

3. Aims and objectives of the Study

The focus of present work is

1. to synthesize efficient, inexpensive, and robust metal oxide electrocatalysts for high Hydrogen/oxygen production efficiencies using soft chemical methods.

2. to characterize them for their structure, morphology and phase purity by using methods various means.

3. and also, to improve the catalytic activity for both oxygen evolution reaction (OER) and Hydrogen evolution reaction (HER) by adding foreign metal ion using in-situ Chemical method.

4. to understand the effect of metal ion doping on performance by various electrochemical Measurements like cyclic –voltametry, Tafel plots, electrochemical impedance Spectroscopy and stability measurement.

5. to understand obtained results metal ion doping on HER/OER Performance computationally.

6. to provide detailed information about the reaction mechanisms involved for OER and HER as a function of metal ion-doping on water splitting application.

7. to compare these results with data reported previously.

8. to write and manuscripts in International Journals of repute.

9. to design a thesis accordingly based on the data available.

4. Methodology

Hydrothermal method

Hydrothermal synthesis is a solution reaction-based approach. In a broader sense, it can be defined as the method for making materials from room temperature to high-temperature solutions. To control the morphology of the materials to be prepared, either low pressure or

high-pressure conditions may be used depending on the vapor pressure of the main composition in the reaction. Many types of nanomaterials have been successfully synthesized by the use of this approach. There are significant advantages of hydrothermal synthesis method over others. Hydrothermal synthesis can generate nanomaterials which are not stable at elevated temperatures. Nanomaterials with high vapor pressures can be produced by the hydrothermal method with minimum loss of materials. The compositions of nanomaterials to be synthesized can be well controlled in hydrothermal synthesis through liquid phase or multiphase chemical reactions. A large number of compounds belonging to practically all classes have been synthesized under hydrothermal conditions: elements, simple and complex oxides, tungstate's, molybdates, carbonates, silicates, germinates etc. Hydrothermal synthesis is commonly used to grow synthetic quartz, gems and other single crystals with commercial value. Some of the crystals that have been efficiently grown are emeralds, rubies, quartz, alexandrite and others. The method has proved to be extremely efficient both in the search for new compounds with specific physical properties and in the systematic physicochemical investigation of intricate multicomponent systems at elevated temperatures and pressures.

The crystallization vessels used are autoclaves. These are usually thick-walled steel cylinders with a hermetic seal which must withstand high temperatures and pressures for prolonged periods of time. Furthermore, the autoclave material must be inert with respect to the solvent. The closure is the most important element of the autoclave. Many designs have been developed for seals, the most famous being the Bridgman seal. In most cases, steel-corroding solutions are used in hydrothermal experiments. To prevent corrosion of the internal cavity of the autoclave, protective inserts are generally used. These may have the same shape as the autoclave and fit in the internal cavity (contact-type insert), or be a "floating" type insert which occupies only part of the autoclave interior. Inserts may be made of carbon free iron, copper silver, gold, platinum, titanium, glass(or quartz),or Teflon, depending on the temperature and solution used.

Fig. 2: Hydrothermal autoclave reactor

5. Characterizations

1) Physical characterizations

a) Structural analysis

Solid materials are formed by atoms or atomic group arranged in certain way. When an x-ray beam is injected into the material (Fig 3), it would be scattered by atoms. If two or more x-ray beams scattered by the atoms that have some phase differences are superimposed onto each other, diffraction is occurred. The x-ray diffraction instrument (SHIMADZU-6000 Model) is used to collect the intensities of the scattered signals to get the diffraction pattern of the measured sample. This pattern is normally as the signal intensity versus the phase angle. When such a pattern is used in the crystal surface calibration process for the sample, the material's crystalline structure, such as the orientation and phase angles, can be obtained. Identification of the phases was made with the help of the Joint Committee on Powder Diffraction Standards (JCPDS) files. An advantage for using X- ray diffraction measurement is that it can analyse the material without causing damages on the material.

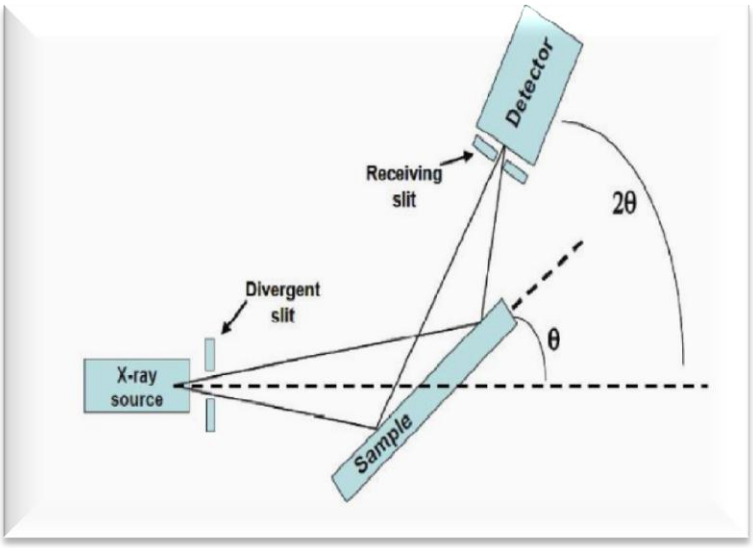

Fig. 3: Ray diagram of X-ray diffractometer

b) Surface moprhology evolution

Scanning Electron Microscopy [SEM] uses a focused electron probe to show structural and chemical information point-by-point from a region of curiosity in the sample

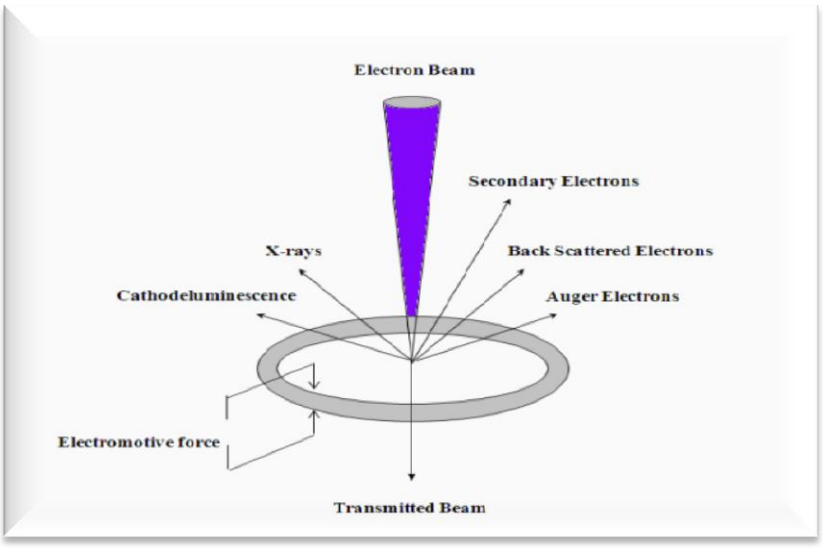

Fig. 4: Different signals produced by electron-sample interactions.

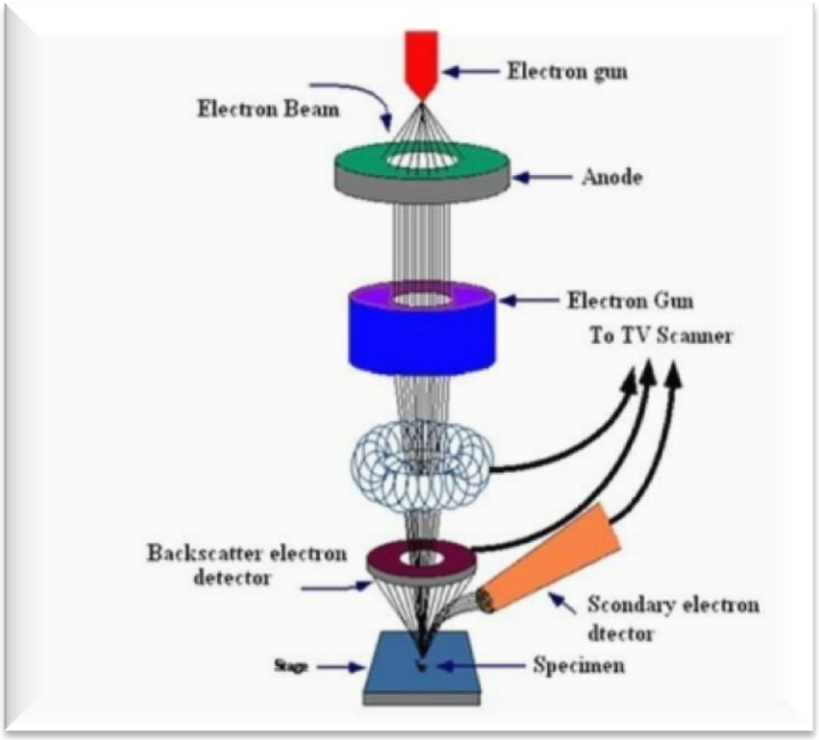

Fig. 5: Ray diagram of scanning electron microscopy.

c) **Raman shift**

Raman spectroscopy is a powerful analytical technique based on inelastic scattering from the sample using a monochromatic light from a laser beam and gives information about transitions between rotational and vibrational states.

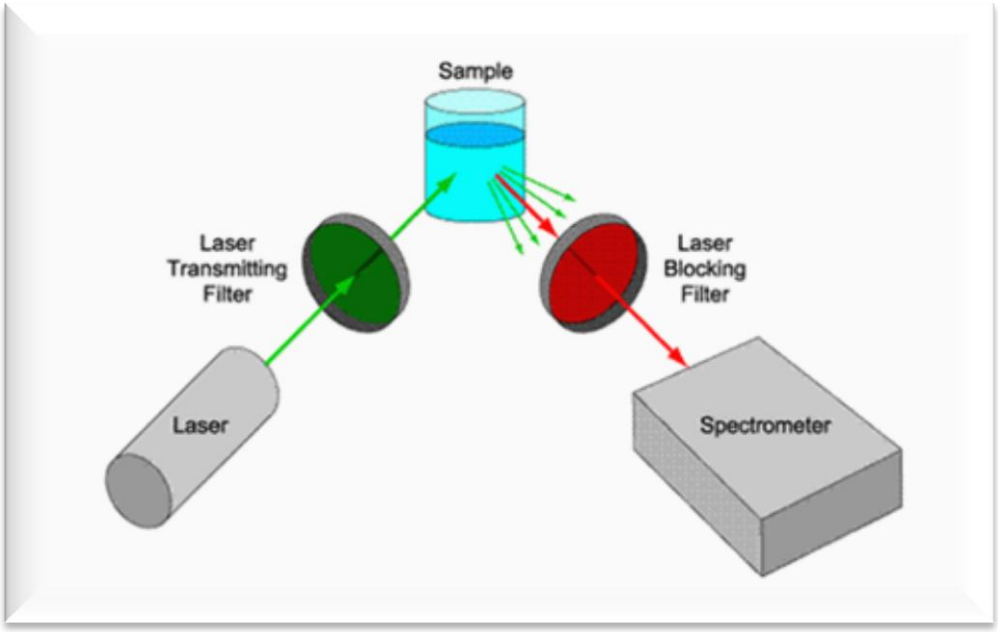

Fig. 6: Schematic diagram of a Raman Spectrometer

d) Transmission electron microscopy measurement

TEM is one of most powerful techniques used in the characterization of nanostructures. TEM is a tool used for lattice imaging and for looking through solid specimens to determine crystalsize and its distribution. TEM generally employs electron beam of extremely high energies between 100 and 300 keV; passing through a thin solid sample. Aschematic diagram shown in Fig 7, a common TEM usually consists of six fundamental components, as follows.

A cathode accelerates the electrons generated from the filament to a high energy, ranging a few hundreds to million volts. Though a higher voltage can produce a higher resolution, actually, the majority TEM instruments are operated at energies between 100 kV and 400 kV. This is to reduce sample injure and the cost of the instrument while still achieving an electron wavelength as short as possible.

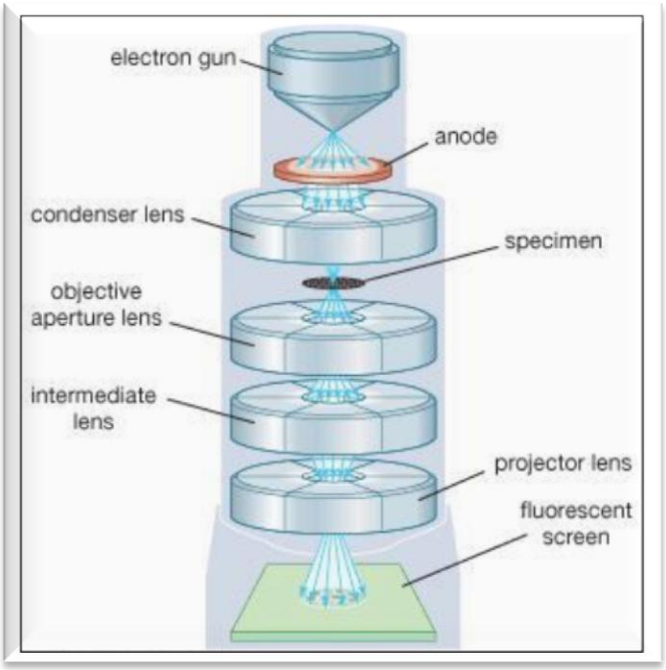

Fig. 7: Schematic diagram of transmission electron microscope.

e) **X-ray photoelectron spectroscopy measurment**

X-ray photoelectron spectroscopy (XPS) is a classical surface-sensitive analytical technique for the surface elemental composition and chemical states information and XPS is a frequently used surface sensitive chemical analysis technique that operates under ultra-high-vacuum.

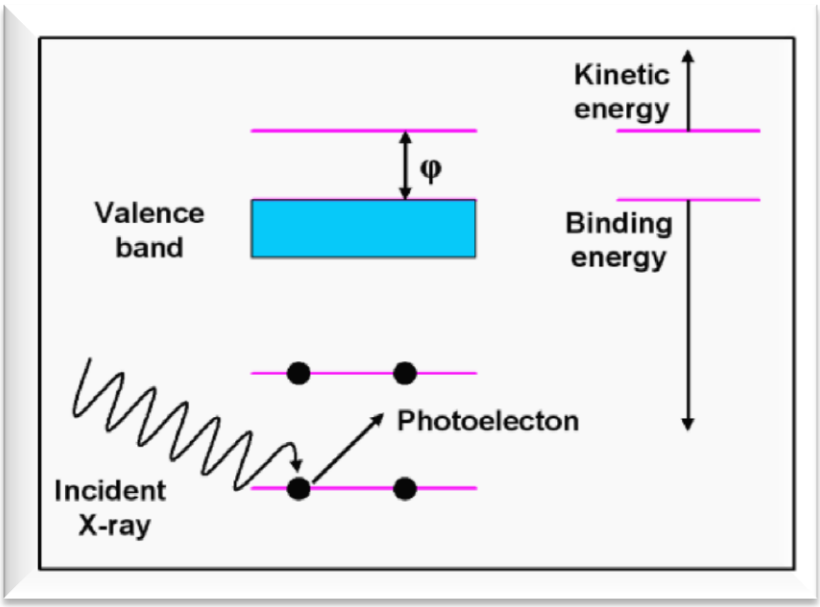

Fig. 8: The mechanism of photoelectron emission in X-ray photoelectron spectroscopy.

5.2 Electrochemical measurements

a) Cyclic voltammetry scanning

Cyclic voltammetry (CV) is a type of potential electrochemical measurement. In a cyclic voltammetry experiment, the working electrode potential is ramped linearly versus time. Unlike in linear sweep voltammetry, after the set potential is reached in a CV experiment, the working electrode's potential is ramped in the opposite direction to return to the initial potential. These cycles of ramps in potential may be repeated as many times as needed. The current at the working electrode is plotted versus the applied voltage (that is, the working electrode's potential) to give the cyclic voltammogram trace. Cyclic voltammetry is generally used to study the electrochemical properties of an analyte in solution or of a molecule that is adsorbed onto the electrode.

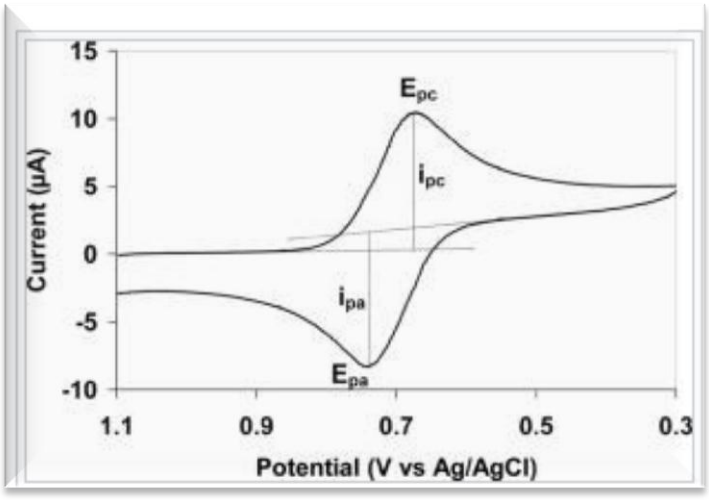

Fig. 9: typical cyclic voltamogram where i_{pc} and i_{pa} Show the cathodic and anodic current respectively for a reversible reaction.

b) Electrochemical impedance spectroscopy

Electrochemical Impedance Spectroscopy (EIS) or ac impedance methods have seen tremendous increase in popularity in recent years. Initially applied to the determination of the charge transfer mechanism and in ac polarography, they are now applied to the characterization of electrode processes and complex interfaces. EIS studies the system response to the application of a periodic small amplitude ac signal. EIS are very often difficult to understand by non-specialists and, frequently, they do not show the complete mathematical developments of equations connecting the impedance with the physico-

chemical parameters. It should be stressed that EIS cannot give all the answers. It is a complementary technique and other methods must also be used to elucidate the interfacial processes.

c) Tafel plot

A Tafel plot is a graphical plot (usually logarithmic) showing the relationship between the current generated in an electrochemical cell and the electrode potential of a specific metal. These plots are usually generated based on electrochemical experiments performed under controlled conditions. And Tafel plots obtained from electrochemical experiments can be used to achieve results in a few hours. Tafel plots are especially useful for predicting corrosion activity in metals or alloys that are highly corrosion resistant.

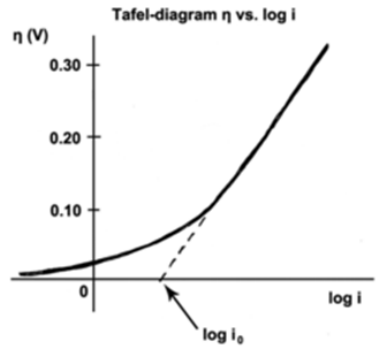

Fig 10: Tafel plot for an anodic process

d) Chemical stability

The stability is another essential parameter to evaluate materials' potential for the practical application. Usually, it can be estimated by cyclic voltammetry (CV) cycling with a high scan rate, which is considered as an accelerate degradation analysis and chronopotentiometric or chronoamperometric test operating at a constant potential or current.

6. Experimental details

Suitable metal oxide electrocatalysts for metal ion–doping water splitting:-

6.1 Synthesis

1. Prepare the sample materials by using the various synthesis methods.

2. In this particular section, attention is paid to the synthetic methods of these HER and OER electrocatalysts, the strategies of performance improvement, and the structure/composition–catalytic activity relationship.

6.2 Physical measurements

In this experimental section firstly, hydrothermal method will be prefered synthesize metal oxides and then physical characterizations of prepared will be measured. It is a fundamental process in the field of material science and nanotechnology, without which no scientific understanding of engineering materials could be ascertained. Characterization is classified on the microscopic, macroscopic, spectroscopic basis. Microscopy is a category of characterization techniques which probe and map the surface and sub-surface of a material some common examples of microscopy instruments.

1. X-ray diffraction analysis (XRD) will be used to identify the single-phase formation of metal oxide/ chalcogenide and its crystal system.

2. The morphology, particle size, grain size and shape are will be demonstrated through advance field emission electron microscopy (FESEM) and transmission electron microscopy (TEM).

6 .3 Electrochemical measurements

For water splitting, all electrochemical measurements were performed in 1 M KOH or Noah (degassed with N_2 for 2 h before measurements). The corresponding current density was normalized to the geometrical area of electrodes. The overall water splitting was performed in a two-electrode configuration, while the half-cell studies were carried out in a three-electrode system, using a Pt. mesh (wire) as a counter electrode, Ag/AgCl (1M or 3M KCl) as a reference electrode and synthesized materials as the working electrode. A glass frit separated the working electrode and the counter electrode. Different techniques were used to quantify the catalytic properties of the prepared electrocatalyst materials. For comparisons, the measured potential was converted to RHE scale, following the equation(1):

$$E_{RHE} = E_{measured} + E^{0}_{Ag/AgCl} + 0.059 \times pH \ (pH = 14 \pm 0.2) \ \ldots\ldots. \ (1)$$

For water electrolysis, the reversible thermodynamic potential against pH when the applied potential is between 0 and 1.23 V, there is no enough driving force to generate O_2 and

H_2. These regions can abeused to investigate the interaction between electrode and electrolyte. When the driving energy is eough to pass the barrier, the gas evolution will start.

1. The electrochemical performances of the indiviual electrodes and fabricated cells will be studied using cyclic voltammetry (CV), galvanostatic charge–discharge analysis (GCD) and impedance spectroscopy (EIS) analysis.
2. In second phase Tofel plot and stability measurement of electrocatalysts will be measured and explored**.**

7. Problem Undertaken

The overpotential and tofel slope of water splitting intimately depend on the electrode materials which we are using. Therefore, further developing new materials with low overpotential and better performance relative to existing electrode materials is the most important technique to overcome these challenges. The best materials can be used as electrode in water splitting is metal oxides/carbon material due to their wide potential window, highly redox reactions, distinct oxidation states, high proton conductivity, remarkably low overpotential, good thermal stability, long cycle life, and high rate capability. In addition, they were also found to be a versatile backbone to composite metal oxides based nanostructures of known value for overpotential. Ternary metal oxides involving offer great promise in the future of electrolysis materials as they can contribute to the good overpotential of the system by an added graphene while the unique combination enable better chemical stability.

8. Plausible outcomes

- High indexed journal research articles will be published.
- Review article based on topic with recent information and advanced is targeted.
- It is also, planed to contribute a book chapter.

9. Index

Chapter 1: Introduction.

Chapter 2: Literature review.

Chapter 3: Metal oxides: Synthesis, characterization and electrochemical properties.

Chapter 4: Effect of Ni-doping on the structure, morphology and electrochemical properties.

Chapter 5: Effect of Co-doping on the structure, morphology, and electrochemical properties.

Chapter 6: Effect of Co-Ni-doping on the structure, morphology and electrochemical properties.

Chapter 7: Results and discussion.

Chapter 8: Summary and Conclusions.

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Research scholar

Ahmed Hasan Hasan Al-naggar

Research Guide

Prof.: R .S. Mane

School of Physical science

S.R.T.M.U. Nanded